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National Bureau of Standards became the National Institute of Standards and Technology on August 23, 1988, when the Omnibus Trade and Competitiveness Act was signed. NIST retains all NBS functions. Its new programs will encourage improved use of technology by U.S. industry.

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**U.S. DEPARTMENT OF COMMERCE**  
**C. William Verity, Secretary**  
**NATIONAL INSTITUTE OF STANDARDS**  
**AND TECHNOLOGY**  
**Ernest Ambler, Director**



## 1. INTRODUCTION

Standard Reference Materials (SRMs) are materials chosen for physical properties which are stable and well characterized. For thermal insulation measurements, SRMs of low thermal conductivity are available having certified values of thermal conductivity over a range of parameters, such as bulk density and mean temperature. SRMs of low thermal conductivity provide a means for accurate inter-laboratory comparison of conductivity measurements. SRMs are utilized by industry, academic, and government laboratories for calibrating heat-flux-meter apparatus, or checking the accuracy of guarded-hot-plate apparatus. New thermal conductivity SRMs are sought to improve the accuracy of these apparatus over a greater operating range.

In 1977, an advisory panel consisting of members from a working group of the American Society of Testing and Materials (ASTM) C-16 Committee developed a plan for selecting materials for SRMs of low thermal conductivity [1]. An SRM was needed having a thermal conductivity of approximately  $0.023 \text{ W/m}\cdot\text{K}$  ( $0.16 \text{ Btu}\cdot\text{in}/\text{h}\cdot\text{ft}^2\cdot^\circ\text{F}$ ), suitable for a temperature range of  $-175$  to  $900^\circ\text{C}$  ( $-280$  to  $1700^\circ\text{F}$ ). The advisory panel decided that microporous fumed-silica insulation was the most suitable material for development of an SRM meeting these requirements.

Recently, four lots of fumed-silica insulation materials were evaluated [2] at the National Bureau of Standards (NBS) in Gaithersburg, Maryland. The materials were evaluated by the following criteria: (1) variability of the thermal conductivity within a lot, (2) variability of material physical properties, (3) relative ease of handling, (4) stability under heat treatment, and (5) economic considerations. One material was recommended as a candidate for further study as a possible SRM.

NBS procured a second lot of candidate material from the manufacturer, Wacker-Chemie GmbH<sup>1</sup>, for further evaluation. Fifteen specimens from the second lot were randomly selected and their thermal conductivity measured using the NBS 1-metre Guarded Hot Plate (GHP) at a mean temperature of  $297 \text{ K}$  and ambient air pressure of  $101.3 \text{ kPa}$ . Thermal conductivity was measured after conditioning each specimen at  $21^\circ\text{C}$  and 30% relative humidity, and then remeasured after being oven-dried at  $100^\circ\text{C}$  and uncontrolled humidity. From the 15 specimens, a smaller set of specimens was measured over the temperature and pressure limits of the apparatus. Results of the evaluation are presented in this report.

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### <sup>1</sup>PRODUCT DISCLAIMER

Because the product is a candidate SRM material, it is necessary to identify the manufacturer so other laboratories may obtain similar material. This in no way represents an endorsement of this material by NBS.

## 2. DESCRIPTION OF SPECIMENS

The specimens of fumed-silica board were obtained in the form of square panels having nominal dimensions of 600 by 600 by 25.4 mm (23.6 x 23.6 x 1 in.). The nominal bulk density for a 600 mm square board was 310 kg/m<sup>3</sup> (19.4 lb/ft<sup>3</sup>). A total of 150 insulation boards was obtained, all manufactured from one batch of raw material.

After inspecting the lot, 75 insulation boards were selected for inclusion in the SRM lot. An additional 15 boards were selected for measurements of thermal conductivity using the NBS 1-metre Guarded Hot Plate. Because of concerns raised about the durability of the material, two boards with surface cracks were selected among the 15 specimens. They were identified as samples B-01 and B-02.

### 2.1 Specimen Material and Manufacturing Process

Fumed-silica insulation board is a silica-aerogel composite comprised primarily of submicron particles of synthetic, amorphous silica (SiO<sub>2</sub>) bonded together in a cellular structure. Information specific to the candidate lot of 150 specimens was obtained from the manufacturer [3]. The fumed-silica insulation material consists of: (1) amorphous silica, 60%; (2) ilmenite (FeTiO<sub>3</sub>), 35%, as an opacifier; and (3) ceramic fiber, 5%, used to strengthen the material. The fumed-silica particles have a surface-area to mass ratio of 300 m<sup>2</sup>/g and a mean-diameter of approximately 10<sup>-8</sup> m.

In the manufacturing process, the raw material is layered between two plates, raked level, then pressed into panel form. The panel is removed and heated at 900°C to burn out all carbonaceous material. The finished product contains no organic compounds.

Previous work [2] showed a small decrease (1 to 2%) in apparent thermal conductivity after heat-treatment at 550°C for eight hours. To prepare the material for testing at high temperatures, the lot of 150 specimens was heat-treated at 650°C by the manufacturer for an additional eight hours.

### 2.2 Material Handling

Compared to fibrous glass board, (NBS SRM 1450b), fumed-silica insulation board is extremely fragile and requires extra care when handling. The material may crack or break if lifted too quickly. At NBS, the specimens were transported by first gently lifting one edge of the specimen and then sliding a flat, smooth support board such as plexiglas or bakelite under the insulation. The support board was then used to transport the insulation. The size of the specimen handled was 600 mm square.

The fumed-silica insulation board is friable. Sliding the support board under the insulation produced a thin layer of "dust" material on the support board. A residual layer of dust material was also deposited on the individual's hands. Gloves were worn when working with the material. As a precaution, NBS personnel also wore dust masks while working with the material. Further information concerning the health issues of fumed-silica is available in ASTM STP 732, Health Effects of Synthetic Silica Particulates [4].

### 3. PRESSURE CONTROL OF THE NBS 1-m GUARDED HOT PLATE

Due to the microporous nature of the material, the apparent thermal conductivity depends not only on the type of gas within the pores of the material, but also the gas pressure. Previous measurements by NBS [2] at atmospheric pressure have shown a variation of 1.5 kPa (15 mbar; 0.44 in. Hg) produced an approximate change of 0.7% in the specimen apparent thermal conductivity. The pressure dependence is caused by the mean-free-path limitation of the gas molecules within the pores of the material.

The original design of the NBS 1-m GHP did not provide for pressure control within the environmental chamber surrounding the apparatus. Thermal measurements of fumed-silica insulation board by NBS [2] were obtained only when the barometric pressure was steady within  $\pm 0.1$  kPa ( $\pm 1$  mbar;  $\pm 0.03$  in. Hg). For this study, a major modification of the apparatus was undertaken to control the ambient air pressure within the environmental chamber.

#### 3.1 Description of the Environmental Chamber

The temperature-controlled plates of the apparatus are housed within a large, six-sided rectangular environmental chamber, 1.4 by 1.4 by 1.6 m high (4.6  $\times$  4.6  $\times$  5.3 ft). The walls of the chamber consist of 150 mm (3 in.) thick panels of polyurethane insulation encased in a structural shell of aluminum sheet reinforced with aluminum cross-members. Two sets of double-doors, both front and back, allow access to the apparatus plates. Air is conditioned and circulated within the chamber by a small heat exchanger consisting of a chilled-water coil, an electric-resistance heater and a blower. Detailed descriptions of the NBS 1-m GHP have appeared previously [5,6]. The apparatus is shown in Figure 1.

#### 3.2 Modifications for Pressure Control

The modifications for controlling air pressure within the environmental chamber required sealing the structure of the chamber as air-tight as possible and installing a controlled air supply and evacuation system. As a starting point, a previous structural analysis

of the environmental chamber by Dr. William Stone [6] was examined to determine the pressure limits the chamber could sustain. The analysis indicated the chamber could withstand an internal pressure of 87.6 to 290 kPa (876 to 2900 mbar; 12.7 to 42 psia) before structural failure could be expected. Initial design considerations were to control the chamber pressure within  $\pm 0.2$  kPa ( $\pm 2$  mbar;  $\pm 0.06$  in. Hg) with a range of  $\pm 1.5$  kPa ( $\pm 15$  mbar;  $\pm 0.44$  in. Hg) around barometric conditions, well within the limits specified in the structural analysis.

In order to locate leaks in the environmental chamber, the doors were removed and replaced with polyethylene sheet taped to the structure walls. The chamber was pressurized slightly and a trace quantity of gaseous refrigerant was injected into the chamber. An electronic leak detector was used to locate escaping refrigerant through the structure. The openings were sealed with silicone room-temperature-vulcanizing rubber.

Several items of hardware on the apparatus were modified to reduce air leakage. Electrical sensor wires and plumbing lines for the cold plates of the apparatus were re-routed through separate openings located in opposing walls of the chamber. Openings around electrical sensor wires were sealed with plastic putty or expanding foam. The plumbing lines were replaced with hard-drawn copper pipe, threaded fasteners and quick disconnects to provide an air-tight seal at the wall interface. The original gasket between the chamber and double-doors was removed and replaced with a miter-cut gasket of natural rubber. Two one-piece aluminum doors were fabricated and replaced the original four double-doors. The new doors were secured to the chamber using four steel brackets clamped at each corner of the door.

Despite these efforts, the chamber was not made completely air-tight. Small leaks persisted around the openings used for electrical sensor wires and between the gasket and doors. These leaks required the air supply and evacuation control system, described below, to be operated continuously during thermal measurement of the specimens.

### 3.3 Air Supply and Evacuation Control System

The air supply and evacuation control system for the environmental chamber is illustrated in Figure 2. A supply air pressure line and an evacuation line provided mass flow ( $m_1$ ) to/from the environmental chamber, compensating for changes in the barometric pressure. Air flow to/from the chamber ( $m_1$ ) was regulated by controlling exhaust/make-up air ( $m_2$ ) with a 3-way motorized valve. A small desk-top microcomputer controlled the valve. A digital pressure transducer produced a feedback signal for the closed-loop control of the 3-way motorized valve.

The supply air system was capable of raising the chamber air pressure 5.5 kPa (55 mbar; 1.6 in. Hg) above ambient atmospheric pressure; the evacuation system, 3.0 kPa (30 mbar; 0.89 in. Hg) below atmospheric pressure. For thermal measurements, however, the systems were limited to

approximately one-half these values. The barometric pressure at the site averaged about 100.0 kPa (1000 mbar) during a five-month test period and ranged between extremes of 98.15 to 102.40 kPa (981.5 to 1024.0 mbar). The majority of the tests were conducted with the environmental chamber pressurized at 101.3 kPa (1 atmosphere at STP). Pressure control within the environmental chamber was measured to be  $\pm 0.2$  kPa ( $\pm 2$  mbar;  $\pm 0.06$  in. Hg).

As mentioned, the small leaks in the structure required operating the air supply and evacuation control system continuously during thermal testing. When supply air was introduced to the chamber, the relative humidity (rh) of the air in the chamber was found to range between 5 and 15%. The evacuation system, when used, caused laboratory air to infiltrate into the chamber. The laboratory air was controlled at 40% rh  $\pm 2\%$  rh.

### 3.4 Uncertainties in the Chamber Pressure Measurement

During thermal testing, the digital pressure transducer measured the chamber air pressure and the corresponding output signal was recorded by the data acquisition system. A precision aneroid barometer and barometric stripchart recorder were used to monitor the barometric pressure. The aneroid barometer was calibrated using the NBS Triple-Scale Aneroid which is reported to be uncertain by  $\pm 10$  Pa ( $\pm 0.10$  mbar;  $\pm 0.003$  in. Hg). The output and display of the pressure transducer were checked with the precision aneroid barometer. The uncertainty of the output of the pressure transducer was estimated to be  $\pm 0.15$  kPa ( $\pm 1.5$  mbar;  $\pm 0.044$  in. Hg). The stripchart recorder was calibrated to within  $\pm 0.05$  kPa ( $\pm 0.5$  mbar;  $\pm 0.01$  in. Hg).

## 4. VARIATION OF SPECIMEN PHYSICAL PROPERTIES

Prior to the measurement of thermal conductivity, the fumed-silica insulation boards were conditioned in laboratory air at  $21^\circ\text{C} \pm 1^\circ\text{C}$ , 30% rh  $\pm 10\%$  rh. Specimens were room-conditioned a minimum of 7 days. Prior to the second set of thermal measurements, the specimens were conditioned for a minimum of 24 hours in an hot-air oven at  $100^\circ\text{C}$ , uncontrolled humidity.

After room conditioning, the thickness of each specimen was measured using a caliper capable of 0.1 mm resolution. The specimen thickness was measured at nine equal-area locations over the 600 mm square specimen and nine measurements were averaged. No significant change was noted in the dimensions of the specimen after conditioning at  $100^\circ\text{C}$ .

The bulk density for the 600 mm square area was determined after conditioning at  $21^\circ\text{C}$ , 30% rh and again after conditioning at  $100^\circ\text{C}$ . The specimen mass was measured with a digital laboratory balance with an

uncertainty of  $\pm 0.1\%$ . Also after conditioning at  $100^\circ\text{C}$ , the moisture content of the room-conditioned specimens was determined.

The average specimen thickness and bulk density for the 15 specimens (samples, B-01 to B-15) are presented in Table 1. The moisture content (by mass) for the room-conditioned specimens is also presented in Table 1. Averages for the three parameters are tabulated and a variation of one standard deviation ( $1\sigma$ ) is included with the average.

The average thickness for the 15 specimens was  $25.7 \text{ mm} \pm 0.1 \text{ mm}$ . The average bulk density for the 15 specimens was  $320.4 \text{ kg/m}^3 \pm 7.1 \text{ kg/m}^3$  after conditioning at  $21^\circ\text{C}$ , 30% rh, and was  $315.2 \text{ kg/m}^3 \pm 6.5 \text{ kg/m}^3$  after conditioning at  $100^\circ\text{C}$ . The average moisture content for the 15 room-conditioned specimens was 1.6%.

## 5. THERMAL CONDUCTIVITY MEASUREMENTS

For brevity, the term, thermal conductivity ( $\lambda$ ), will be used to denote apparent thermal conductivity in this report. The thermal conductivity of the fumed-silica insulation specimens was measured using the NBS 1-metre Guarded Hot Plate. The measurements examined the effect of bulk density ( $\rho$ ), moisture content (MC), mean temperature ( $T_m$ ), and barometric pressure (P), on the thermal conductivity.

Tests were performed in accordance with procedures described in ASTM test method C-177 and practice C-1044 [7,8]. For all tests, the guarded hot plate was operated in a one-sided mode of operation. The backflow specimen was aged, extruded polystyrene, 150 mm (6 in.) thick. In general, steady-state test conditions were achieved within 24 hours.

The thermal conductivity of the specimen was determined by the following equation;

$$\lambda = L/R \quad (1)$$

where L is the average specimen thickness, (m), and R is the thermal resistance of the specimen, ( $\text{K}\cdot\text{m}^2/\text{W}$ ), defined as the ratio of temperature difference ( $\Delta T$ ) to time-rate heat flux ( $\dot{Q}/A$ ). The average specimen thickness (L) was described above. An uncertainty of  $\pm 0.8\%$  was estimated for the thickness measurement. The uncertainty in the thermal resistance measurement was estimated to be  $\pm 0.5\%$  [9]. Assuming the two uncertainties to be equally probable in both directions, an overall uncertainty of  $\pm 0.9\%$  was estimated for the thermal conductivity measurement.

A low-density fibrous-glass batt having a nominal thickness of 90 mm (3.5 in.) was used as the mask material. The thermal conductivity of the fibrous-glass batt compressed to 25.48 mm (1.003 in.) was found to be 0.0339 W/m $\cdot$ K. The effect on the specimen heat transfer was checked by

stacking and measuring two oven-dried specimens, samples B-07 and B-11. The thermal resistance of the two stacked specimens was within 0.3% of the sum of the individual resistances (at 297 K, 101.3 kPa).

## 6. ANALYSIS OF DATA

### 6.1 Dependence on Bulk Density

Thermal conductivity measurements for the 15 specimens conditioned at 21°C, 30% rh are summarized in Table 2. Measurements were conducted at a mean specimen temperature of 297 K and chamber air pressure of 101.3 kPa. The initial and final moisture contents (MC) by mass for each specimen are also summarized. All specimens experienced a loss of mass during the thermal conductivity measurement as shown in Table 2. The mass loss was attributed to migration of water vapor from the specimen during the test period.

Thermal conductivity measurements for the 15 specimens conditioned 24 hours at 100°C and uncontrolled humidity are summarized in Table 3. Measurements were conducted at a mean specimen temperature of 297 K and chamber air pressure of 101.3 kPa. The final moisture content for each specimen indicates all specimens experienced a small increase in moisture content during the test period.

Thermal conductivity measurements as a function of bulk density are illustrated in Figure 3 for the data from Tables 2 and 3. Measurements for each specimen are shown as individual data points. The circular data points are for specimens room-conditioned and the square points are for specimens oven-dried prior to thermal measurement. A linear least-squares fit for each set of data was obtained using the NBS statistical program Dataplot [10]. The best-fit equation is of the following form;

$$\lambda = A_0 + A_1 \cdot \rho \quad (2)$$

where,  $A_0$  and  $A_1$  are regression coefficients and  $\rho$  is the specimen bulk density ( $\text{kg}/\text{m}^3$ ). Regression coefficients are presented below for the two sets of data.

Curve A (room-conditioned):	$\lambda = 0.01323 + 2.592 \times 10^{-5} \cdot \rho$
Curve B (oven-dried):	$\lambda = 0.01462 + 1.992 \times 10^{-5} \cdot \rho$

A moisture content of 1.6% (by mass) increased the specimen thermal conductivity of the fumed-silica insulation by approximately 2.4% at 297 K, 101.3 kPa.

Calculated residuals (in percent) for the Curve "B" equation are illustrated in Figure 4. For 99% confidence limits ( $\pm 3\sigma$ ), the departure from the fit is within  $\pm 1.1\%$ . For the room-conditioned data, the departure from Curve "A" is within  $\pm 1.7\%$  at 99% confidence limits.

## 6.2 Reproducibility of the Thermal Conductivity Measurement

Two thermal conductivity measurements for two room-conditioned specimens, samples B-08 and B-15, are summarized in Table 2. The two measurements for samples B-08 and B-15 were within 3.3% and 0.3%, respectively, of each other. Different specimen moisture contents at the time of measurement were responsible for the large variation of conductivity for sample B-08.

Three different thermal conductivity measurements for sample B-09 are summarized in Table 3. For each measurement, the specimen was oven-dried at 100°C a minimum of 24 hours prior to measurement. The thermal conductivity measurements at test conditions of 297 K, 101.3 kPa were reproducible to within ±0.4%.

## 6.3 Temperature Dependence

Thermal conductivity measurements for sample B-08 at three mean temperatures and an average chamber air pressure of 101.2 kPa are summarized in Table 4. Measurements were conducted at mean temperatures of 283.11, 297.00 and 311.02 K and chamber air pressures of 101.08, 101.39, 101.26 kPa, respectively.

Thermal conductivity measurements as a function of mean specimen temperature are illustrated in Figure 5 for the data from Table 4. A linear equation was fit to the data. The final form of the best-fit equation is presented below;

$$\lambda = 0.01650 + 1.612 \times 10^{-5} \cdot T_m$$

where  $T_m$  is the mean specimen temperature in K.

## 6.4 Pressure Dependence

Thermal conductivity measurements for several specimens at different chamber air pressures and  $T_m$  at 297 K are summarized in Tables 5 and 6. Measurements in Table 5 are for room-conditioned specimens. Measurements in Table 6 are for specimens conditioned at 100°C.

Thermal conductivity measurements as a function of chamber air pressure for specimens with three or more data points are illustrated in Figure 6. Measurements are shown as individual data points. Samples B-11 and B-15 were room-conditioned. Samples B-01, B-06, B-09, B-13, B-14 and B-11 again, were conditioned at 100°C. A linear least-squares fit (solid line) was obtained for the data for each specimen.

Regression pressure coefficients are summarized in Table 7. While the intercept term ( $A_0$ ) varies for each specimen due to different bulk density and moisture contents, the slope term ( $A_1$ ) for each specimen is similar. The average slope for all specimens is  $7.540 \times 10^{-5}$  (W/m·K·kPa) with a variation of one standard deviation ( $1\sigma$ )  $\pm 0.455 \times 10^{-5}$  (W/m·K·kPa).

## 7. DISCUSSION

### 7.1 Thermal Characterization

The thermal conductivity of specimens from the candidate lot of fumed-silica insulation board is characterized for the ranges of bulk density, mean temperature and barometric pressure shown in Table 8, below.

**Table 8.** Range of parameters for thermal characterization of the fumed-silica insulation board.

<u>Parameter</u>	<u>Range</u>	<u>Units</u>
Bulk Density - Dry	$304.5 < \rho < 325.4$	(kg/m <sup>3</sup> )
Mean Temperature	$283.11 < T_m < 311.02$	(K)
Barometric Pressure	$97.51 < P < 103.43$	(kPa)

Thermal conductivity measurements on some representative specimens from these lots at lower pressures as well as high temperatures are in process at NBS-Boulder, Colorado (USA) [11].

### 7.2 Results of Thermal Measurements

The low variation,  $\pm 1.1\%$  at 99% confidence limits, for the oven-dried specimen thermal conductivity as a function of bulk density is considered excellent. Measurement reproducibility for the oven-dried specimens is also considered excellent.

The variation for thermal conductivity measurements of room-conditioned specimens is also excellent,  $\pm 1.7\%$  (99% confidence limits). However, the measurements of room-conditioned specimens were not always reproducible. Observe the difference between thermal conductivity measurements of sample B-08 in Table 2. Different moisture contents at the time of measurement were responsible for the large variation of conductivity for sample B-08. In fact, the moisture content of all the room-conditioned specimens varied (see Table 2), even though the conditioning time was about the same, one to two weeks. For this reason, NBS recommends the SRM material be measured at room-temperature only after receiving adequate drying, such as heat-treatment at 100°C for 24 hours prior to measurement.

A change of 27.9 K in mean temperature produced a change of 2.1% in the specimen thermal conductivity (see Table 4).

A change of 3.6 kPa in the chamber air pressure around ambient atmospheric pressure caused a change of 1.3% in the specimen thermal conductivity (see Table 5, sample B-15). The effect of ambient pressure on the thermal conductivity of other NBS SRMs (fibrous-glass board and

blanket, SRMs 1450b and 1451, respectively) is only observed at much lower ambient pressure, below 10 kPa (100 mbar) [12]. For thermal conductivity apparatus without pressure control, measurement of this fumed-silica SRM will require recording the barometric pressure. Fortunately, the barometric pressure can remain stable (within  $\pm 0.1$  kPa) for several hours.

### 7.3 Variation of Atmospheric Pressure with Altitude

The change in atmospheric pressure due to altitude is examined using the NACA standard atmosphere relationships [13]. For altitudes below 10,769 m (below the isothermal layer), the following relationship is appropriate;

$$P = P_0 \cdot \left[ 1 - \left( \frac{a}{T_0} \right) Z \right]^{(aR)^{-1}} \quad (3)$$

where,

- $P_0$  = Standard pressure at sea level = 101.325 kPa = 760 mm Hg
- $a$  = Standard temperature lapse rate =  $0.0065 \text{ K} \cdot \text{m}^{-1}$
- $T_0$  = Standard absolute temperature at sea level = 288 K
- $Z$  = Altitude (m)
- $R$  = Standard gas constant for dry air =  $29.2745 \text{ m} \cdot \text{K}^{-1}$

The low ambient pressure limit achieved for the NBS 1-m GHP was 97.51 kPa (975.1 mbar; 28.88 in. Hg). For  $P = 97.51$  kPa, Equation 3 yields an upper elevation limit of 322.4 m (1058 ft). For reference only, an expanded listing of values for the NACA standard atmosphere is provided in the Appendix, Table A1.

### 7.4 Durability

The fumed-silica insulation is a relatively fragile material for use as an SRM. Surface cracks will eventually develop on a specimen and propagate with repeated usage. To ascertain the effect of surface cracks on the thermal conductivity, two specimens, samples B-01 and B-02, were specifically selected with cracks on the specimen. The crack in sample B-01 ran the entire length of the specimen, passing through the area corresponding to the metered-area of the 1-m GHP. No discernable difference in specimen thermal conductivity was noted for either of these specimens (see Tables 2 and 3). However, to avoid mechanical damage and extend the lifetime of the SRM, NBS recommends using a smooth support board when transporting the material.

## 7.5 Moisture and Liquid Penetration Concerns

The microporous fumed-silica insulation is hygroscopic and should be stored in an area with a relative humidity of 50% or less. Direct contact with liquids should be avoided. Contact with liquids causes irreparable damage to the material in the area of contact. The affected area experiences permanent shrinkage.

## 8. CONCLUSIONS

Thermal conductivity measurements were completed on 15 specimens from a second lot of fumed-silica insulation using the NBS 1-metre Guarded Hot Plate. The measurements examined the effect of bulk density, mean temperature, barometric pressure, and moisture content on the specimen thermal conductivity. The material is characterized for the following ranges of these parameters: bulk density, 304.5 to 325.4 kg/m<sup>3</sup>; mean temperature, 283.1 to 311.0 K; and barometric pressure, 97.51 to 103.43 kPa. The effect of moisture content on room-temperature measurement can be minimized by conditioning the specimen at 100°C for 24 hours prior to measurement. This material is sufficiently characterized at room-temperature to be offered as a Standard Reference Material of low thermal conductivity. Seventy-five samples (600 by 600 by 25.4 mm) of the fumed-silica insulation were transferred to the Office of Standard Reference Materials in Gaithersburg, Maryland, USA.

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## APPENDIX

TABLE A1

NACA Standard Atmosphere, Lower Atmosphere (Expanded Listing)

<u>Elevation</u>		<u>Pressure</u>				
(m)	(ft)	(kPa)	(mbar)	(mm Hg)	(in Hg)	(atm)
-100	-328	102.533	1025.33	769.06	30.28	1.012
0	0	101.325	1013.25	760.00	29.92	1.000
100	328	100.129	1001.29	751.03	29.57	0.988
200	656	98.944	989.44	742.14	29.22	0.977
300	984	97.771	977.71	733.34	28.87	0.965
400	1312	96.609	966.09	724.63	28.53	0.953
500	1640	95.458	954.58	716.00	28.19	0.942
600	1969	94.319	943.19	707.45	27.85	0.931
700	2297	93.190	931.90	698.98	27.52	0.920
800	2625	92.073	920.73	690.60	27.19	0.909
900	2953	90.966	909.66	682.30	26.86	0.898
1000	3281	89.870	898.70	674.08	26.54	0.887
1100	3609	88.785	887.85	665.94	26.22	0.876
1200	3937	87.710	877.10	657.88	25.90	0.866
1300	4265	86.646	866.46	649.90	25.59	0.855
1400	4593	85.593	855.93	642.00	25.28	0.845
1500	4921	84.550	845.50	634.17	24.97	0.834
1600	5249	83.517	835.17	626.43	24.66	0.824
1700	5577	82.494	824.94	618.76	24.36	0.814
1800	5906	81.482	814.82	611.16	24.06	0.804
1900	6234	80.479	804.79	603.65	23.77	0.794
2000	6562	79.487	794.87	596.20	23.47	0.784

TABLE 1

Physical properties of the fumed-silica specimens.  
 Specimens conditioned at 21°C, 30% rh and then oven-dried at 100°C.  
 Moisture content is for specimens conditioned at 21°C, 30% rh.

Sample Number	Average Specimen Thickness (mm)	Bulk Density		Moisture Content by mass (%)
		Room Conditioned (kg/m <sup>3</sup> )	Oven Dried (kg/m <sup>3</sup> )	
B-01	25.8	328.4	322.5	1.8
B-02	25.7	317.2	310.6	2.1
B-03	25.7	327.3	322.2	1.6
B-04	25.8	325.3	319.3	1.9
B-05	25.6	314.2	309.9	1.4
B-06	25.7	310.5	306.1	1.5
B-07	25.5	318.9	313.5	1.7
B-08	25.5	333.1	325.4	2.4
B-09	25.6	313.8	309.4	1.4
B-10	25.6	308.5	304.5	1.3
B-11	25.7	318.1	313.6	1.4
B-12	25.7	319.9	315.1	1.5
B-13	25.7	318.6	312.8	1.9
B-14	25.5	328.1	323.2	1.5
B-15	25.7	324.1	319.8	1.3
Average	25.7	320.4	315.2	1.6
Standard Deviation	±0.1	±7.1	±6.5	±0.3

TABLE 2

Thermal conductivity measurements at  $T_m = 297$  K,  $P = 101.3$  kPa.  
 Specimens conditioned at  $21^\circ\text{C}$ , 30% rh.  
 MC = moisture content.

Sample Number	Average Specimen Thickness (mm)	Bulk Density ( $\text{kg/m}^3$ )	Plate Temperatures		Measured Thermal Conductivity ( $\text{W/m}\cdot\text{K}$ )	Initial MC (%)	Final MC (%)	Mass Change (g)
			Hot (K)	Cold (K)				
-01	25.8	328.4	310.90	283.13	0.02161	1.8	1.7	-4.5
-02	25.7	317.2	310.93	283.10	0.02167	2.1	1.9	-7.8
-03	25.7	327.3	310.95	283.07	0.02155	1.6	1.5	-3.8
-04	25.8	325.3	310.95	283.10	0.02188	1.9	1.7	-5.0
-05	25.6	314.2	310.97	283.11	0.02144	1.4	1.3	-2.7
-06	25.7	310.5	310.92	283.09	0.02123	1.5	1.4	-1.0
-07	25.5	318.9	310.96	283.12	0.02154	1.7	1.7	-2.0
-08	25.5	333.1	310.96	283.11	0.02202	2.4	2.1	-7.5
-08*	25.5	330.3	310.92	283.07	0.02131	1.2	1.1	-5.2
-09	25.6	313.8	310.97	283.10	0.02141	1.4	1.3	-4.4
-10	25.6	308.5	310.94	283.11	0.02121	1.3	1.0	-7.8
-11	25.7	318.1	310.93	283.12	0.02136	1.4	1.3	-3.2
-12	25.7	319.9	310.94	283.11	0.02143	1.5	1.4	-3.4
-13	25.7	318.6	310.93	283.12	0.02139	1.9	1.6	-6.7
-14	25.5	328.1	310.93	283.10	0.02169	1.5	1.4	-3.4
-15	25.7	324.1	310.96	283.10	0.02159	1.3	1.2	-4.2
-15*	25.7	324.2	310.95	283.11	0.02152	1.4	1.1	-7.3

Specimen remeasured

TABLE 3

Thermal conductivity measurements at  $T_m = 297$  K,  $P = 101.3$  kPa.  
 Specimens conditioned at  $100^\circ\text{C}$ , uncontrolled humidity (24 hr minimum).  
 MC = moisture content.

Sample Number	Average		Plate Temperatures		Measured Thermal Conductivity (W/m·K)	Initial MC (%)	Final MC (%)	Mass Change (g)
	Specimen Thickness (mm)	Bulk Density (kg/m <sup>3</sup> )	Hot (K)	Cold (K)				
B-01	25.8	322.5	310.95	283.11	0.02106	0	0.3	+10.0
B-02	25.7	310.6	310.97	283.12	0.02084	0	0.3	+ 8.8
B-03	25.7	322.2	310.97	283.12	0.02103	0	0.3	+ 9.4
B-04	25.8	319.3	310.94	283.11	0.02116	0	0.3	+ 9.6
B-05	25.6	309.9	310.96	283.12	0.02090	0	0.3	+ 9.1
B-06	25.7	306.1	310.95	283.15	0.02073	0	0.4	+10.9
B-07	25.5	313.5	310.96	283.11	0.02082	0	0.3	+ 8.4
B-08	25.5	325.4	310.97	283.13	0.02097	0	0.3	+10.2
B-09	25.6	309.4	310.96	283.11	0.02070	0	0.4	+12.2
B-09*	25.6	309.4	310.95	283.12	0.02084	0	0.4	+10.9
B-09*	25.6	309.3	310.95	283.10	0.02080	0	0.4	+10.4
B-10	25.6	304.5	310.94	283.11	0.02070	0	0.3	+ 7.9
B-11	25.7	313.6	310.96	283.11	0.02078	0	0.3	+ 9.3
B-12	25.7	315.1	310.94	283.09	0.02089	0	0.4	+10.2
B-13	25.7	312.8	310.95	283.11	0.02079	0	0.4	+12.7
B-14	25.5	323.2	310.95	283.13	0.02113	0	0.3	+ 9.4
B-15	25.7	319.8	310.98	283.12	0.02102	0	0.3	+ 9.4

\* Specimen remeasured

TABLE 4

Thermal conductivity measurements at  $P = 101.2$  kPa,  $T_m$  varies.  
Specimen conditioned at  $21^\circ\text{C}$ , 30% rh.

Sample Number	Average Specimen Thickness (mm)	Bulk Density ( $\text{kg/m}^3$ )	Plate Temperatures			Measured Thermal Conductivity ( $\text{W/m}\cdot\text{K}$ )	Chamber Air Pressure (kPa)
			Hot (K)	Cold (K)	Mean (K)		
B-08	25.5	330.3	297.12	269.11	283.11	0.02106	101.08
B-08	25.5	330.3	310.92	283.07	297.00	0.02131	101.39
B-08	25.5	330.3	325.10	296.94	311.02	0.02151	101.26

TABLE 5

Thermal conductivity measurements at  $T_m = 297$  K, P varies.  
Specimens conditioned at 21°C, 30% rh.

Sample Number	Average Specimen Thickness (mm)	Bulk Density (kg/m <sup>3</sup> )	Plate Temperatures		Measured Thermal Conductivity (W/m·K)	Chamber Air Pressure (kPa)
			Hot (K)	Cold (K)		
B-11	25.7	318.1	310.94	283.12	0.02116	98.55
B-11	25.7	318.1	310.94	283.12	0.02117	98.83
B-11	25.7	318.1	310.93	283.12	0.02136	101.40
B-11	25.7	318.1	310.93	283.12	0.02125	99.97
B-11	25.7	318.1	310.95	283.12	0.02137	101.03
-----						
B-15	25.7	324.1	310.89	283.10	0.02156	100.48
B-15	25.7	324.1	310.96	283.10	0.02159	101.25
B-15	25.7	324.1	310.96	283.10	0.02145	99.36
B-15	25.7	324.1	310.96	283.11	0.02165	101.89
B-15	25.7	324.1	310.97	283.10	0.02147	99.69
-----						
B-15 *	25.7	324.2	310.95	283.11	0.02152	101.24
B-15	25.7	324.2	310.96	283.11	0.02142	99.86
B-15	25.7	324.2	310.95	283.11	0.02160	102.02
B-15	25.7	324.2	310.95	283.12	0.02169	103.40
B-15	25.7	324.2	310.95	283.11	0.02170	103.43
B-15	25.7	324.2	310.95	283.11	0.02154	101.48
B-15	25.7	324.2	310.96	283.12	0.02146	100.39
B-15	25.7	324.2	310.96	283.12	0.02145	100.30
-----						
B-15 *	25.7	324.2	310.96	283.12	0.02135	99.34
B-15	25.7	324.2	310.96	283.12	0.02128	98.43
B-15	25.7	324.2	310.96	283.12	0.02140	100.03

\* Specimen remeasured

TABLE 6

Thermal conductivity measurements at  $T_m = 297$  K, P varies.  
Specimens conditioned at 100°C, uncontrolled humidity, 24 hr minimum.

Sample Number	Average Specimen Thickness (mm)	Bulk Density (kg/m <sup>3</sup> )	Plate Temperatures		Measured Thermal Conductivity (W/m·K)	Chamber Air Pressure (kPa)
			Hot (K)	Cold (K)		
B-01	25.8	322.5	310.96	283.11	0.02095	99.77
B-01	25.8	322.5	310.96	283.11	0.02089	98.96
B-01	25.8	322.5	310.95	283.11	0.02106	101.29
B-01	25.8	322.5	310.96	283.11	0.02096	99.99
B-01	25.8	322.5	310.96	283.11	0.02095	99.85
-----						
B-02	25.7	310.6	310.98	283.12	0.02078	100.33
B-02	25.7	310.6	310.97	283.12	0.02084	101.26
-----						
B-03	25.7	322.2	310.97	283.12	0.02094	99.97
B-03	25.7	322.2	310.97	283.12	0.02103	101.26
-----						
B-05	25.6	309.9	310.92	283.11	0.02080	99.96
B-05	25.6	309.9	310.96	283.12	0.02090	101.34
-----						
B-06	25.7	306.1	310.96	283.14	0.02067	100.57
B-06	25.7	306.1	310.95	283.15	0.02073	101.25
B-06	25.7	306.1	310.97	283.15	0.02044	97.82
B-06	25.7	306.1	310.97	283.15	0.02041	97.51
-----						
B-08	25.5	325.4	310.97	283.13	0.02097	101.25
B-08	25.5	325.4	310.97	283.12	0.02090	100.45
-----						
B-09	25.6	309.4	310.97	283.12	0.02048	98.12
B-09	25.6	309.4	310.96	283.11	0.02061	99.87
B-09	25.6	309.4	310.96	283.11	0.02070	101.26
-----						
B-09 *	25.6	309.4	310.96	283.11	0.02070	98.98
B-09	25.6	309.4	310.96	283.11	0.02068	98.71
B-09	25.6	309.4	310.95	283.12	0.02084	101.16
-----						
B-11	25.7	313.6	310.96	283.11	0.02073	100.59
B-11	25.7	313.6	310.96	283.11	0.02078	101.24
B-11	25.7	313.6	310.96	283.11	0.02065	99.52
-----						
B-13	25.7	312.8	310.95	283.11	0.02077	100.99
B-13	25.7	312.8	310.95	283.10	0.02080	101.32
B-13	25.7	312.8	310.95	283.10	0.02082	101.59
B-13	25.7	312.8	310.95	283.11	0.02081	101.45
B-13	25.7	312.8	310.95	283.11	0.02079	101.13
-----						
B-14	25.5	323.2	310.96	283.11	0.02107	100.51
B-14	25.5	323.2	310.95	283.13	0.02113	101.25
B-14	25.5	323.2	310.96	283.12	0.02098	99.33

\* Specimen remeasured

TABLE 7.

Pressure regression coefficients for room-conditioned  
and oven-dried specimens.

Sample Number	$A_0$ (W/m·K)	$A_1$ (W/m·K·kPa)
<u>Room-conditioned</u>		
B-11	0.01341	$7.852 \times 10^{-5}$
B-15	0.01380	$7.697 \times 10^{-5}$
B-15 *	0.01350	$7.932 \times 10^{-5}$
B-15 *	0.01408	$7.320 \times 10^{-5}$
<u>Oven-dried</u>		
B-01	0.01340	$7.566 \times 10^{-5}$
B-06	0.01226	$8.362 \times 10^{-5}$
B-09	0.01356	$7.055 \times 10^{-5}$
B-09 *	0.01407	$6.701 \times 10^{-5}$
B-11	0.01319	$7.492 \times 10^{-5}$
B-13	0.01347	$7.233 \times 10^{-5}$
B-14	0.01330	$7.727 \times 10^{-5}$
Average		$7.540 \times 10^{-5}$
Standard Deviation		$\pm 0.455 \times 10^{-5}$

\* Specimen remeasured

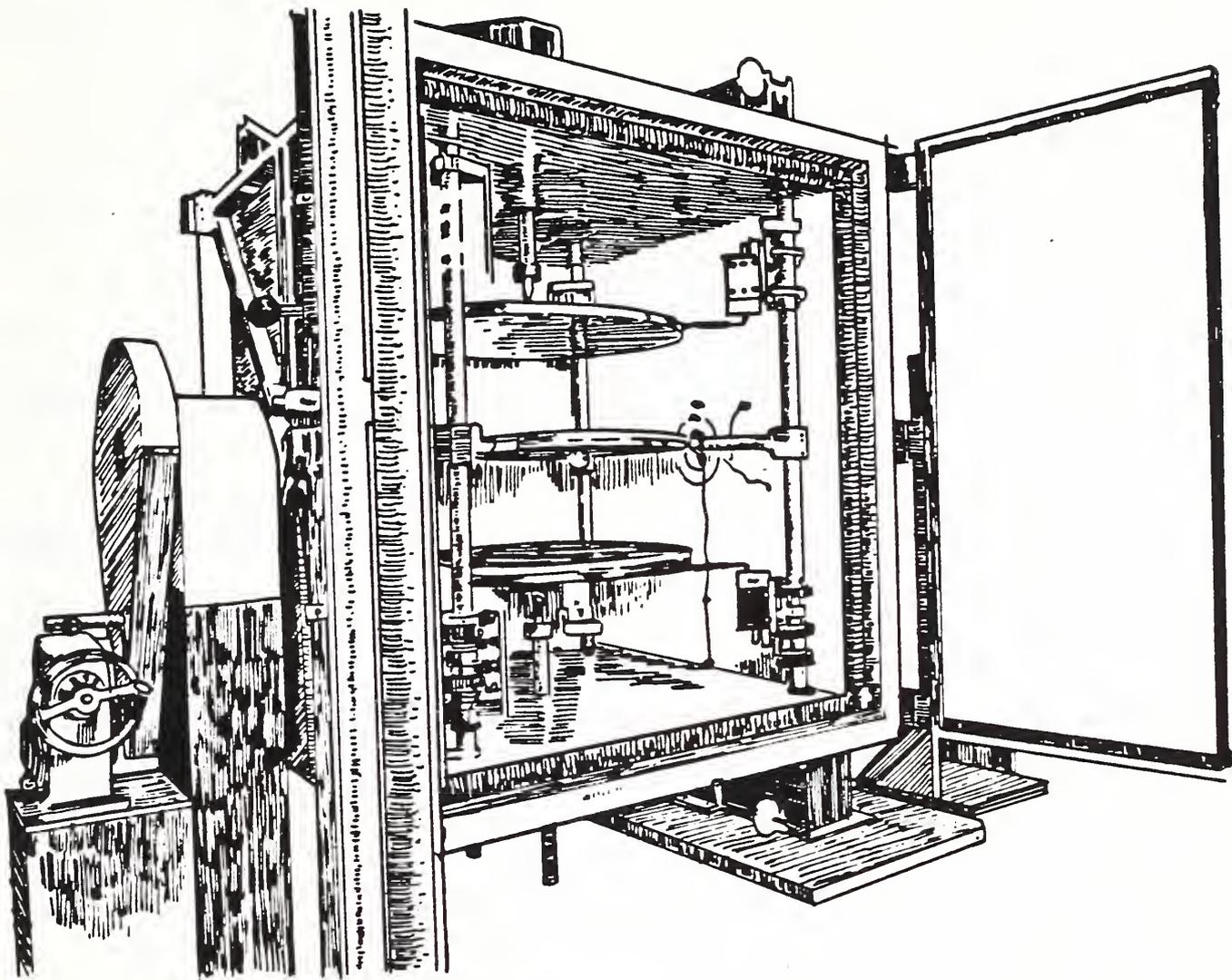


Figure 1. Diagram illustrating the National Bureau of Standards 1-metre Guarded Hot Plate.

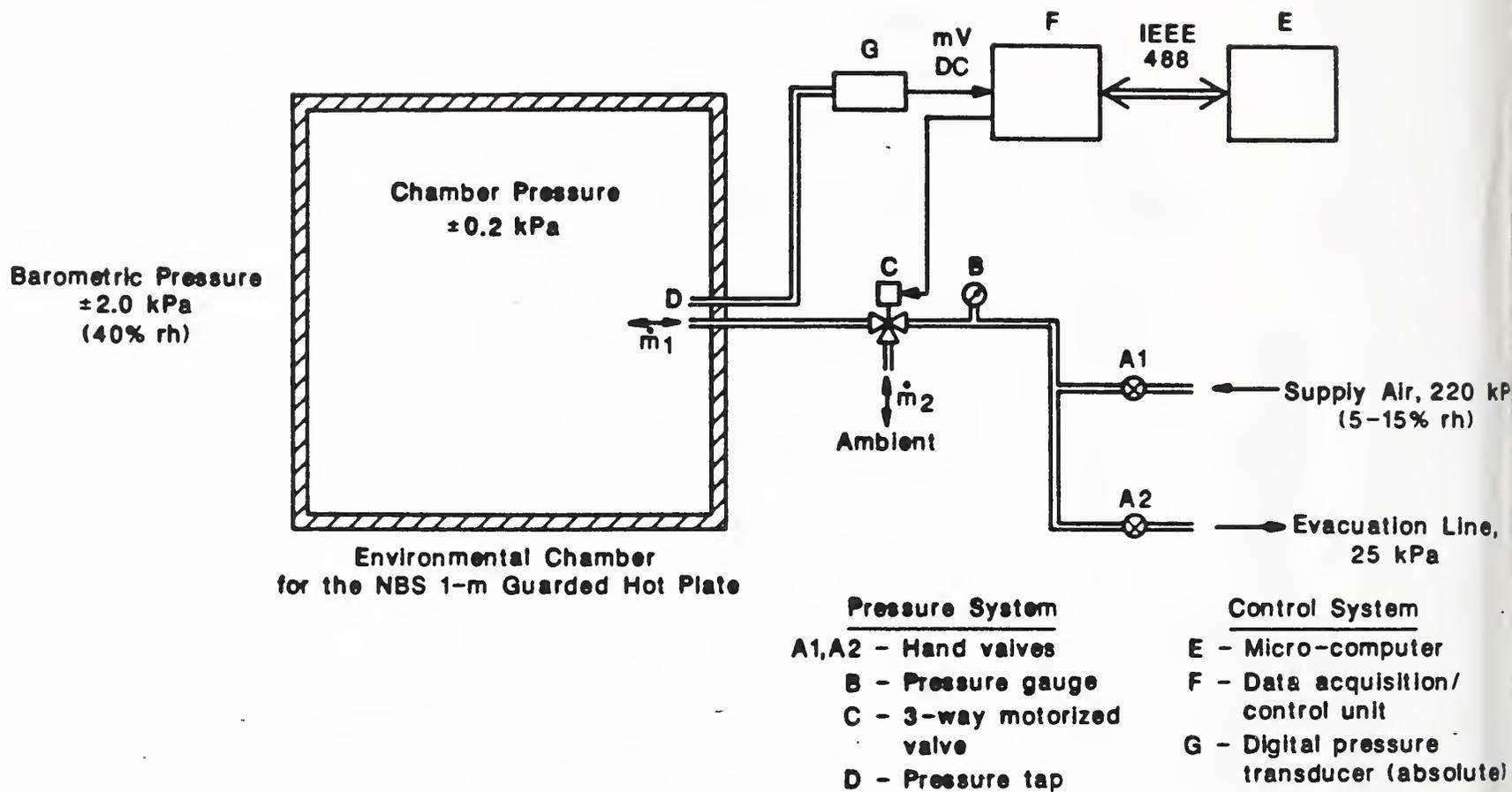


Figure 2. Schematic diagram of the air supply and evacuation control system for the NBS 1-metre Guarded Hot Plate.

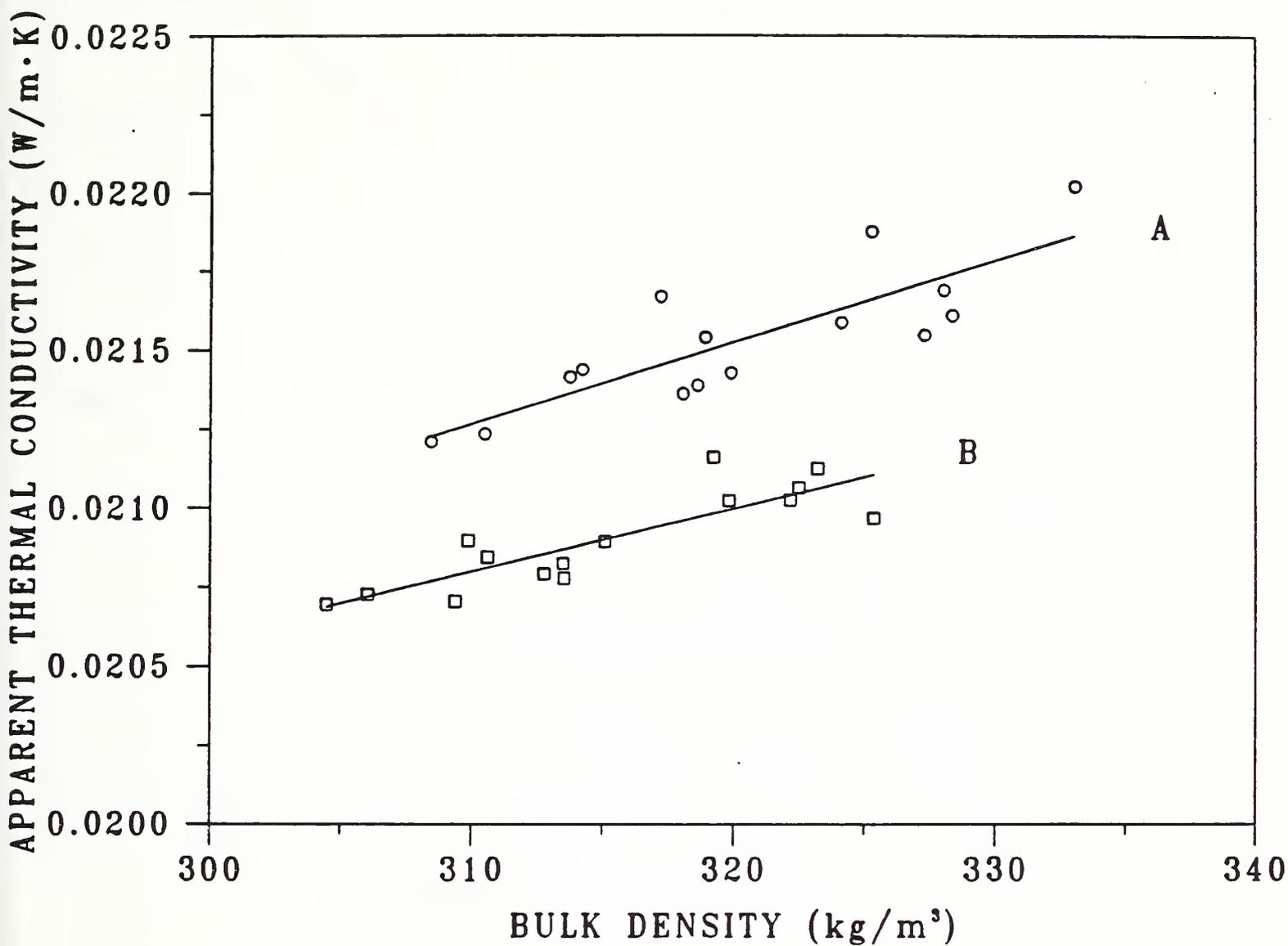


Figure 3. Thermal conductivity at  $T_m = 297$  K and  $P = 101.3$  kPa as a function of specimen bulk density for fumed-silica insulation. Curve "A" is the best fit for specimens conditioned at  $21^\circ\text{C}$ , 30% rh (average specimen moisture content 1.6%). Curve "B" is for specimens oven-dried at  $100^\circ\text{C}$ .

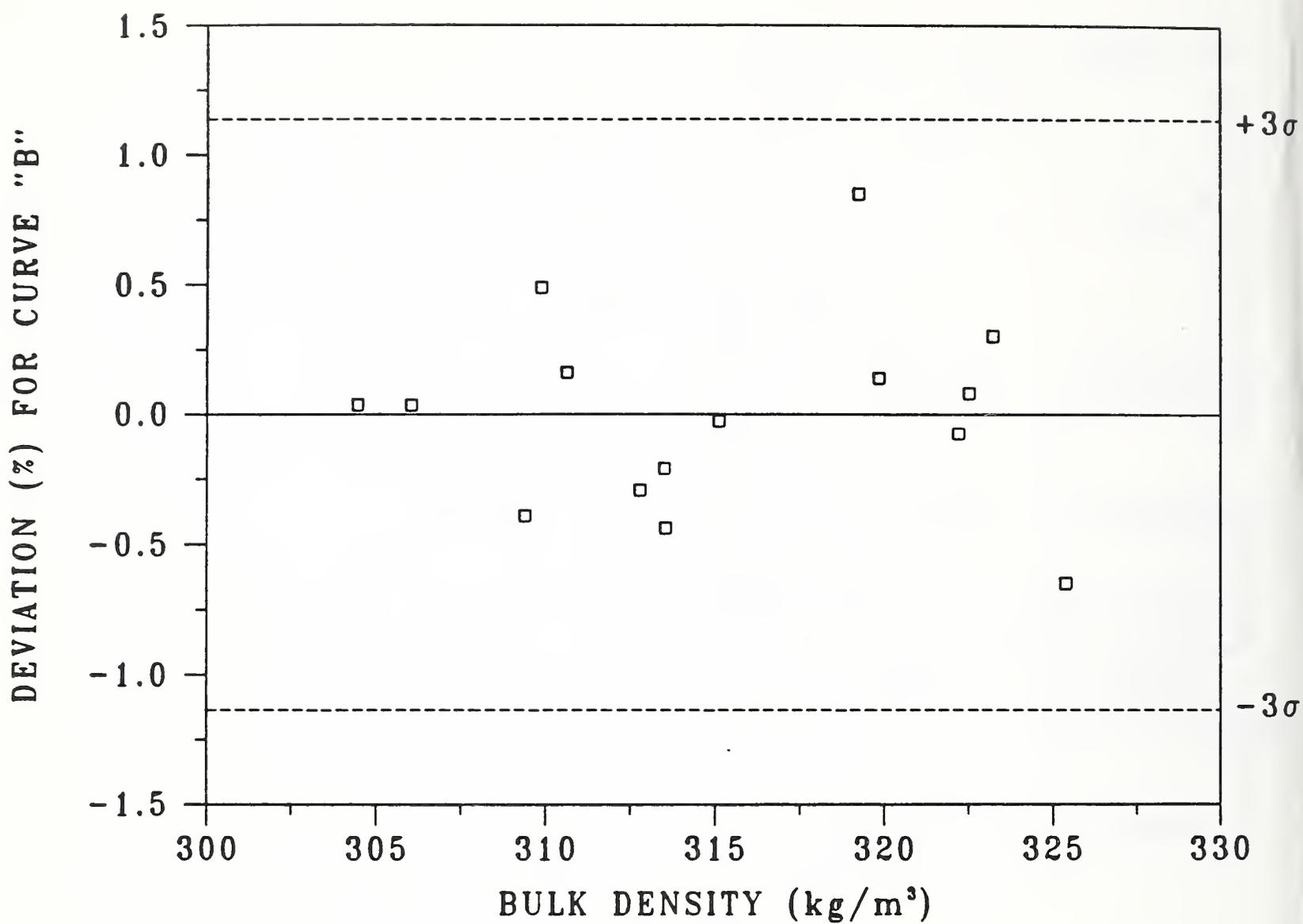


Figure 4. Calculated residuals (in percent) for the best-fit curve for the oven-dried specimens (Curve "B"). All residuals are within 99% confidence limits ( $\pm 3\sigma$ ).

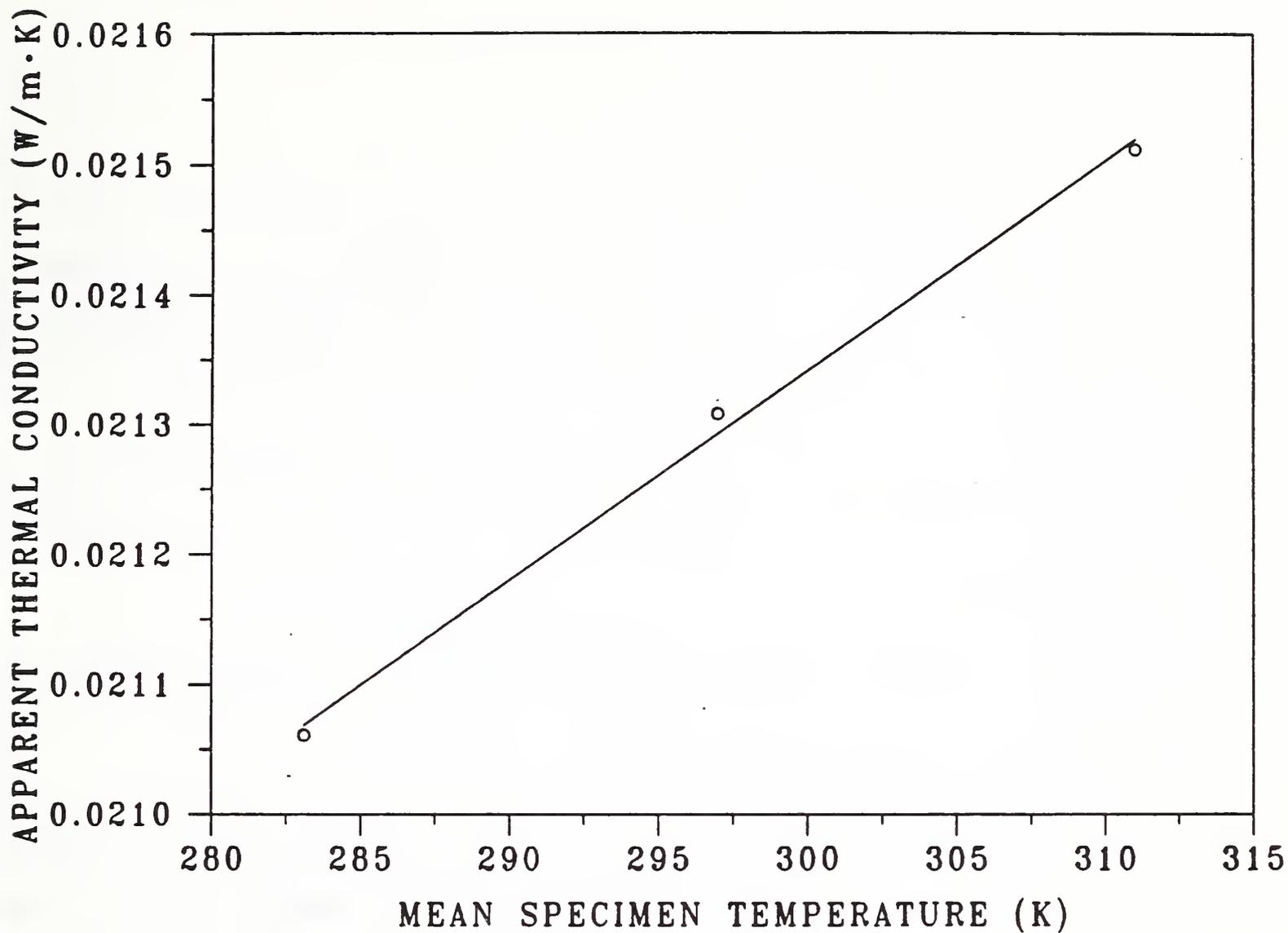


Figure 5. Thermal conductivity at  $P = 101.3$  kPa as a function of mean specimen temperature for fumed-silica insulation.

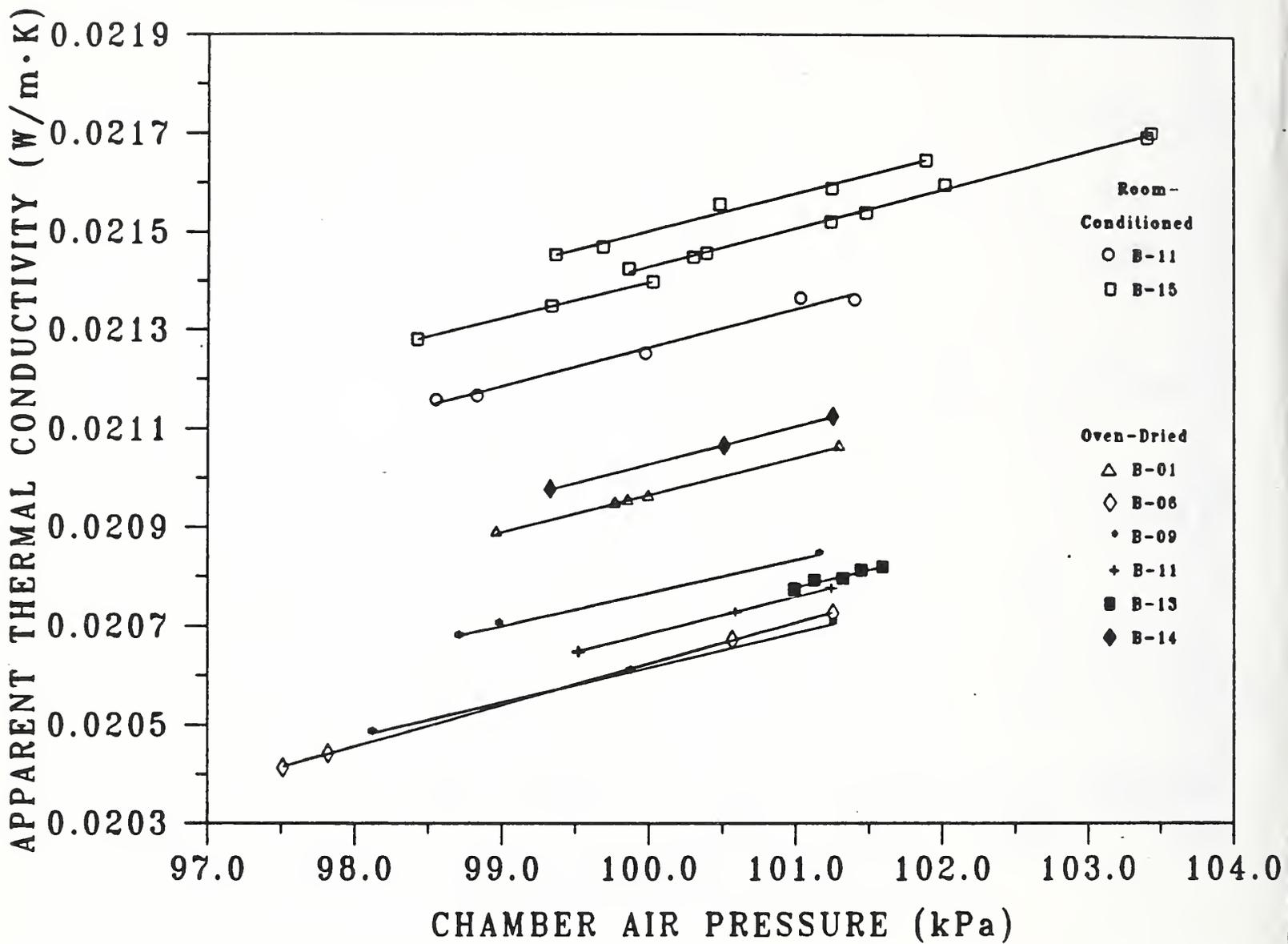


Figure 6. Thermal conductivity at  $T_m = 297$  K as a function of chamber air pressure for fumed-silica insulation.

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**TITLE AND SUBTITLE**  
 Room-Temperature Thermal Conductivity of Fumed-Silica Insulation for A Standard Reference Material

**AUTHOR(S)**  
 Robert R. Zarr, Thomas A. Somers, Donn F. Ebberts

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**9. SUPPLEMENTARY NOTES**  
  
 Document describes a computer program; SF-185, FIPS Software Summary, is attached.

**10. ABSTRACT** (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)  
 Thermal conductivity of fumed-silica insulation board was measured using the National Bureau of Standards 1-metre Guarded Hot Plate. Measurements were conducted for the following range of parameters: bulk density, 304.5 to 325.4 kg/m<sup>3</sup>; mean temperature, 283.1 to 311.0 K; and barometric pressure, 97.51 to 103.40 kPa. The effect of moisture content on room-temperature measurements was minimized by prior conditioning of the specimen at 100°C for 24 hours. Seventy-five samples (600 by 600 by 25.4 mm) were transferred to the Office of Standard Reference Materials in Gaithersburg, Maryland, USA. The material is offered as a Standard Reference Material having a low thermal conductivity at room-temperature.

**12. KEY WORDS** (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)  
 apparent thermal conductivity; fumed-silica insulation; guarded-hot-plate apparatus; Standard Reference Material

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